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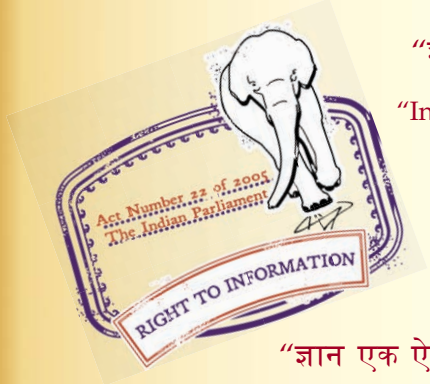

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Indian Standard

METHODS OF SAMPLING AND TEST FOR FERTILIZERS

Part 1 SAMPLING

(First Revision)

UDC 631·813 : 620·113

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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September 1985

Indian Standard

METHODS OF SAMPLING AND TEST FOR FERTILIZERS

Part 1 SAMPLING

(First Revision)

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AMENDMENT NO. 1 MAY 2003
TO
IS 6092 (Part 1) : 1985 METHODS OF SAMPLING AND TEST FOR FERTILIZERS
PART 1 SAMPLING

(First Revision)

(*Page* 4, *clause* **2.1.3**) — Insert a new clause after **2.1.3** and renumber the subsequent clauses:

2.1.4 In case of high density polyethylene packing and also when the fertilizer material is not in free flowing condition, the use of sampling probe may not be possible. In such a case, selected bags for drawing samples may be opened and the fertilizers may be taken out of the bags and spread on a clean surface and samples drawn with the help of a suitable sampling device which may be made of stainless steel or brass cup.

(*Page* 6, *clause* **3.1.3.3**) — Insert a new clause after **3.1.3.3**:

'**3.1.4** In case of chelated micro-nutrients and mixtures of micro-nutrients, the three identical containers of the batch, grade, type and manufacturer, shall be selected which shall constitute the composite samples, provided it is not possible to draw a composite sample of the size given below:

Weight of one sample — One sample of fertilizer shall have the approximate weight, as specified below:

i)	For straight micro-nutrient fertilizers	– 100 g
ii)	For chelated micro-nutrient fertilizers and mixture of micro-nutrients	50 g or the maximum packing size of similar quantity
iii)	For other fertilizers and mixtures of fertilizers	400 g

(*Page* 7, *Table* 1) — Substitute the following Table 1 for the existing:

Table 1 Number of Containers or Packages to be Selected for Sampling
(*Clause* 3.1.3.2)

Lot Size *N*	No. of Bags Selected for Sampling *n*
Less than 10	1
10 to 100	2
101 - 200	3
201 - 400	4
401 - 600	5
601 - 800	6
801 - 1 000	7
1 001 - 1 300	8
1 301 - 1 600	9
1 601 - 2 000	10

(*Page* 12, *clause* 3.4.1.3) — Insert following new clauses after clause 3.4.1.3:

3.5 Sampling from Big Godown/High Stacking

If the procedure given in 3.1.3.2 is not possible to be adopted, the sample should be drawn from the randomly selected fertilizer bags from different layers, from top and from all open sides in a zig zag fashion.

3.6 Sampling from Small Godown

All the fertilizer bags of the same grade and type of each manufacturer though received on different dates shall be segregated and properly stacked. All bags of the same grade and type of fertilizer manufactured by a particular manufacturing unit may be considered as one lot based on their physical conditions and the sample shall be drawn as per procedure laid down in clauses 3.1.2, 3.1.3.2, 3.1.4 and 3.7.

3.7 Sampling from Damaged Stock

a) In case of torn or lumpy bags, damaged fertilizer bags or sweepings, the stock should be arranged according to identifiable lots. From each lot the number of bags shall be selected as per procedure in Table 1. If the bags allow the use of sampling probe conveniently, the samples should be drawn by sampling probe.

b) In case it is not possible to use the sampling probe, the bags may be opened and fertilizer material mixed together uniformly by hammering the big lumps or putting pressure, if required, and then samples drawn by using

(PCD 20)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 NOVEMBER 2003
TO
IS 6092 (PART 1) : 1985 METHODS OF SAMPLING AND TEST FOR FERTILIZERS

PART 1 SAMPLING

(First Revision)

[*Amendment No.* 1, *page* 2, *clause* 3.7(b)] — Substitute the following for the existing matter:

'b) In case it is not possible to use the sampling probe, the bags may be opened and fertilizer material mixed together uniformly by hammering the big lumps or putting pressure, if required, and then samples drawn by using suitable sample device'.

(*Page* 16, *clause* **5.6**) — Substitute the following for the existing clause:

'**5.6** Roll sample slowly from four directions until sample has been thoroughly mixed (5 times is usually considered adequate). This ensures complete mixing. Trying to roll rapidly will cause a sliding.'

(PCD 20)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHODS OF SAMPLING AND TEST FOR FERTILIZERS

Part 1 SAMPLING

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 1) (First Revision) was adopted by the Indian Standards Institution on 25 February 1985, after the draft finalized by the Fertilizers Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1971. Taking into consideration the views of the manufacturers, testing authorities and the technologists; the latest equipment used in the fertilizer trade, industry and in analytical methods, the Sectional Committee decided to revise the standard.

0.3 In the present revision the sampling procedures for bulk fertilizers and for anhydrous ammonia have been included.

0.4 In the preparation of this standard, consideration has been given, to the need for maintaining coordination with the methods of sampling and test for fertilizers prescribed under the *Fertilizer (Control) Order 1957* and the *Essential Commodities Act, 1955* (incorporating amendment up to August 1981) of the Government of India. However, this standard is subject to the provisions imposed under this Order wherever applicable.

0.5 In the preparation of this standard, considerable assistance has been derived from the following publications:

Official Methods of Analysis, The Association of Official Analytical Chemists, Washington, USA, 1978.

Fertilizer Sampling and Analytical Methods. The Fertilizer Institute Product Quality Committee, Washington, USA, 1974.

Recommended Analytical Methods of the National Plant Food Institute. Washington, USA.

BS 4431-1969 Methods of test for anhydrous ammonia. British Standards Institution, UK.

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 The standard (Part 1) prescribes the methods of drawing test samples from bags, as also of drawing samples of bulk material from wagon or truck loads or from flowing streams from a transfer belt or spout and from heaps or piles. It also covers the methods of sampling for anhydrous ammonia from pressurized containers.

2. GENERAL REQUIREMENTS OF SAMPLING OF SOLID FERTILIZERS

2.1 In drawing samples the following precautions and directions shall be observed.

2.1.1 Samples shall be taken from the place of storage, but the process of sampling shall not unnecessarily be exposed to weather.

2.1.2 The sampling equipments shall be clean, dry and should not be a source of contamination.

2.1.3 The material being sampled, the sampling equipments and the containers for samples shall be protected from adventitious contamination. Metallic containers must be avoided.

2.1.4 To draw a representative sample from bagged fertilizers, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

2.1.5 The sample immediately after being drawn shall be placed in wide-mouthed clean, dry and air-tight glass or other suitable containers for example PVC or polyethylene, which must not react with the sample.

2.1.6 The sample containers shall be of such a size that they are almost completely filled by the sample (but not tightly packed).

2.1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and other important particulars of the consignment.

*Rules for rounding off numerical values (*revised*).

2.1.8 For micronutrients and impurities, the equipments made from materials like plastic, aluminium, and wood shall be used. Stainless steel equipments if used must be of the best quality free from any scratches.

2.1.9 Samples shall be stored in shade under dry weather conditions.

3. SAMPLING

3.1 Sampling from Bagged Fertilizers

3.1.1 *Apparatus*

3.1.1.1 *Slotted single tube probe* — (*see* Fig. 1). Solid cone tip constructed of stainless steel or brass. Unplated brass for samples on which micronutrients are to be determined must not be used.

The length of the probe, exclusive of handle, should approximately be equal to the diagonal of the bag sampled. As fertilizers are normally marketed in 50 kg bags in India, an overall length of 110 cm would be adequate for fertilizers of even low bulk density. A slot length of 105 cm and a width of 1·5 cm would be appropriate. The inside diameter of the probe should be 1·5 cm minimum.

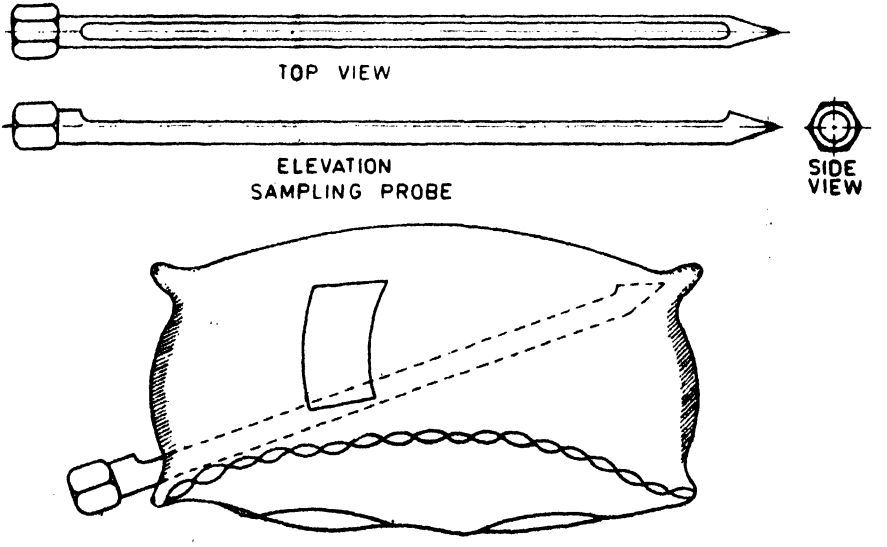


FIG. 1 SAMPLING TECHNIQUE FROM FERTILIZER BAG USING SLOTTED SINGLE TUBE PROBE

3.1.2 *Procedure*

a) Place the bag in a horizontal position, then roll or flip over three or more times.

b) Open the bag by cutting a couple of stitches at one corner and insert the probe so that it extends diagonally from corner to corner (*see* Fig. 1) with slot down. It should not pierce through the bag. Turn it one half turn to bring the slot up, jar bag slightly to fill the probe, and remove carefully so as not to drag material out of it with the bag edges.

c) Empty entire contents of each probe into a suitable container. Take one core per bag. Combine contents of all probes of the lot being sampled and place in a container with moisture seal. Label the contents before another lot is sampled.

d) In case the material in the bag is in a caked condition the bag is dropped several times from a height about 1 metre till lumps are all broken up before sampling with the probe. Mild strokes with a wooden hammer may be helpful. In cases of extreme difficulty, the bag may be opened out and a sample taken either by riffling or by coning and quartering.

e) A double tube probe as prescribed under sampling of bulk material can also be used for sampling bags.

3.1.3 *Scale of Sampling*

3.1.3.1 *Lot* — All containers in a single cansignment of the material of the same grade and type, coming out from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches or sources of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots. In the case of a consignment drawn from a continuous process, 2 000 bags (or 100 tonnes) of the material shall constitute a lot.

3.1.3.2 The number of containers to be chosen for sampling from a lot shall depend on the size of the lot and shall be as given in Table 1.

3.1.3.3 These containers must be selected at random from the lot. In order to ensure randomness of selection, reference may be made to IS : 4905-1968*. In case this standard is not available, the following procedure may be adopted:

Starting from any container in the lot, count them as 1, 2, 3,....., up to r and so on, r being the integral part of N/n. Every rth container thus counted shall be withdrawn till the requisite number of containers are obtained.

*Methods for random sampling.

TABLE 1 NUMBER OF CONTAINERS OR PACKAGES TO BE SELECTED FOR SAMPLING

(*Clause* 3.1.3.2)

Lot Size	No. of Containers to be Selected
N	*n*
(1)	(2)
2 to 8	2
9 to 27	3
28 to 64	4
65 to 100	5
101 to 300	6
301 to 500	7
501 to 800	8
801 to 1 300	9
1 301 and above	10

3.2 Sampling from Bulk Fertilizers

3.2.1 *Double Tube Probe* (*Missouri 'D' Tube*) — For sampling bulk fertilizer in trucks, trailors, wagons or storage bins, the double probe shall comply the requirements as given in Table 2 (*see* Fig. 2).

Note — The single tube probe as prescribed for bags is unsuitable for the purpose, so double tube probe should be used.

TABLE 2 REQUIREMENTS OF DOUBLE TUBE PROBE

All dimensions in millimetres.

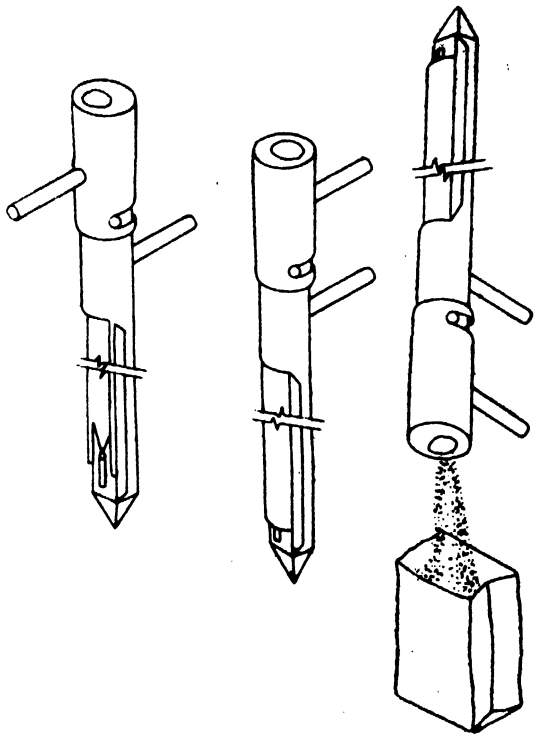
Length	Outer Diameter	Inner Diameter	Compartments	
			No.	Size
1 300	30	25	1	1 100

3.2.2 *Stream Sampling Cup from Belt Samples* — Fertilizer from belts or chutes may be sampled as it drops from the belt or from a chute by using the stream sampling cup (*see* Fig. 3 and 4). Inside dimensions of the cup mouth are 1·9 × 25·4 cm. The length must be as long as maximum diameter of stream. For case of supporting the cup and carrying out the operation, a track of two steel rods may be mounted in the path of, and at right angles to the stream flow. Suitable guides attached to the cup and a handle placed on the side permit use of this device.

3.3 Sampling from Bulk Material in Stores

3.3.1 *Procedure*

3.3.1.1 It is satisfactorily applicable for sampling coned, ridged, flat or one-sided piles containing up to 100 tonnes of material. It is not



STEP 1 STEP 2 STEP 3

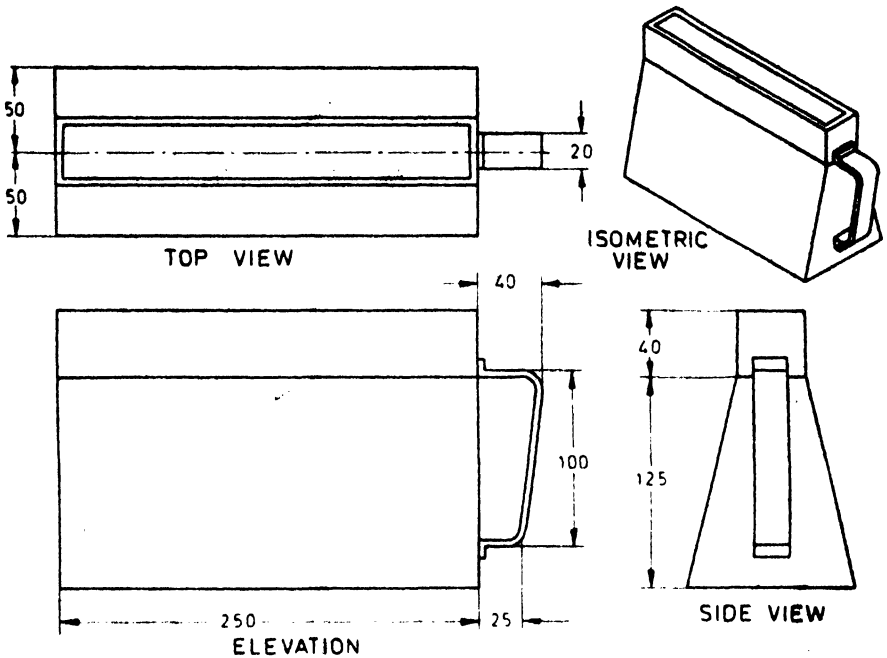
STEP 1 Insert trier (Missouri 'D' Tube) vertically to desired depth in open position.
STEP 2 Bring 'T' handles together to close trier and withdraw.
STEP 3 Pour core withdrawn directly in sample container.

FIG. 2 DOUBLE TUBE PROBE (MISSOURI 'D' TUBE)

possible to accurately sample larger piles with the official sampling devices.

3.3.1.2 The double tube probe is inserted vertically to its full depth in the open position at the locations indicated in Fig. 5 and 6 for undivided trucks and piles respectively. It is closed and then withdrawn. Level or flat piles, containing up to 100 tonnes, are sampled in a fashion similar to that given for the ridged piles. Withdraw 10 cores (to the maximum possible depth of the probe) from locations indicated as shown in Fig. 5 using Missouri 'D' Tube.

3.3.1.3 The 10 probe samples from different points are composited, riffled and prepared for analysis as given under **4** and **5**. If a probe is not available, draw samples (10 samples minimum) for each heap or wagon by scoop from different parts and at different depths so that the sample taken is representative of the bulk.



All dimensions in millimetres.

FIG. 3 STREAM SAMPLING CUP

3.3.1.4 A one-sided or sloped pile is sampled at the points located as shown in Fig. 6A and 6B. Withdraw one vertical core of material from locations 1 and 6, and two cores at locations 2, 3, 4 and 5. Composite all the probe samples, riffle and prepare the sample for analysis.

3.3.1.5 These sampling patterns are so designed that core samples taken from each location represent approximately equal fractions of the lot.

3.4 Sampling from Belt or Spout

3.4.1 *Procedure*

3.4.1.1 For sampling fertilizer material falling from a transfer belt or spout, use the stream sampling cup (*see* Fig. 4) passing it completely through the stream of material as it drops down from a transfer belt or spout. The long slot in the top of the sampling cup should be perpendicular to the falling stream. Pass the cup through the complete stream at a uniform speed, such that the cup will collect approximately equal amounts from each pass, but will never overflow. Empty contents of the cup from each pass into a suitable container.

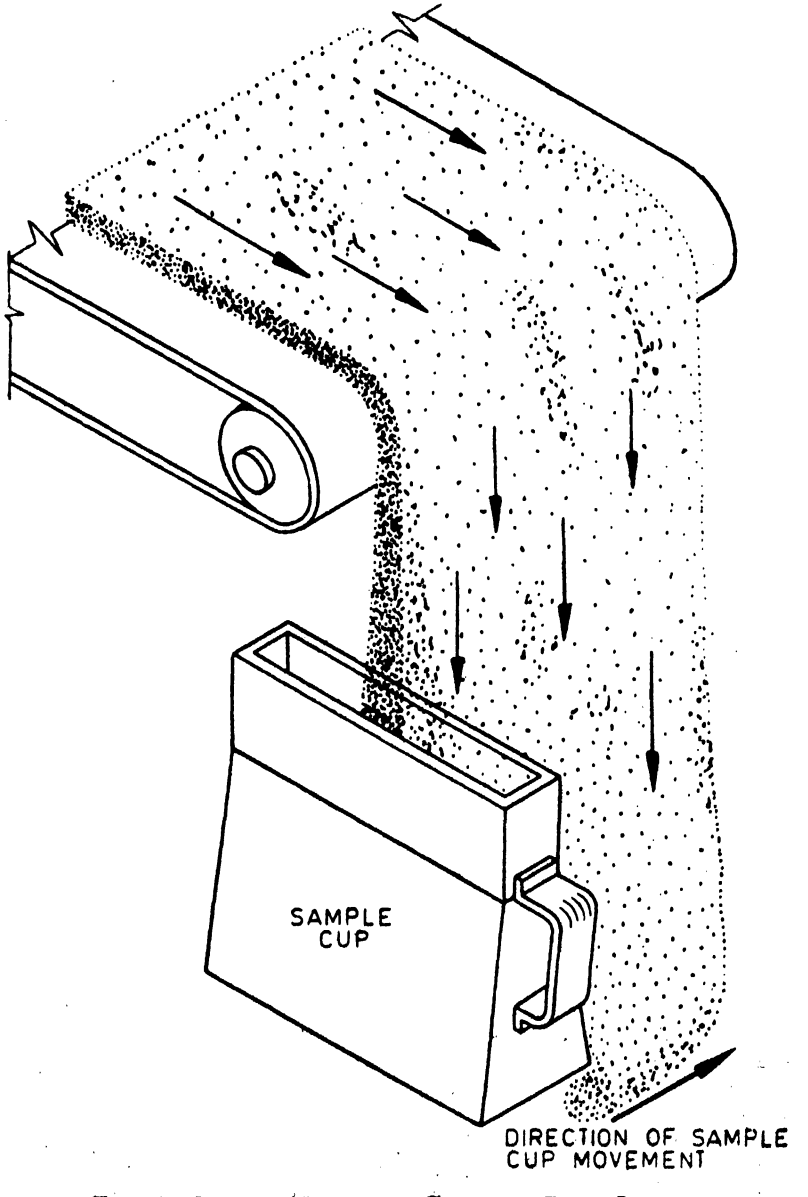


FIG. 4 STREAM SAMPLING CUP FOR BELT SAMPLES

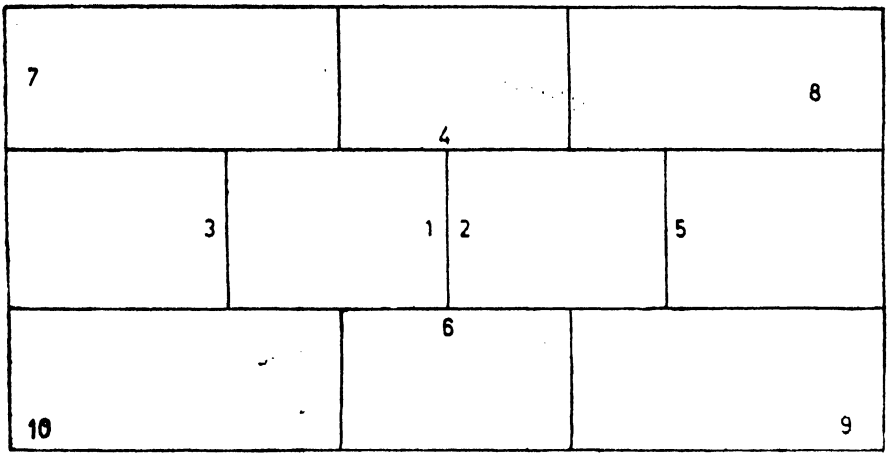
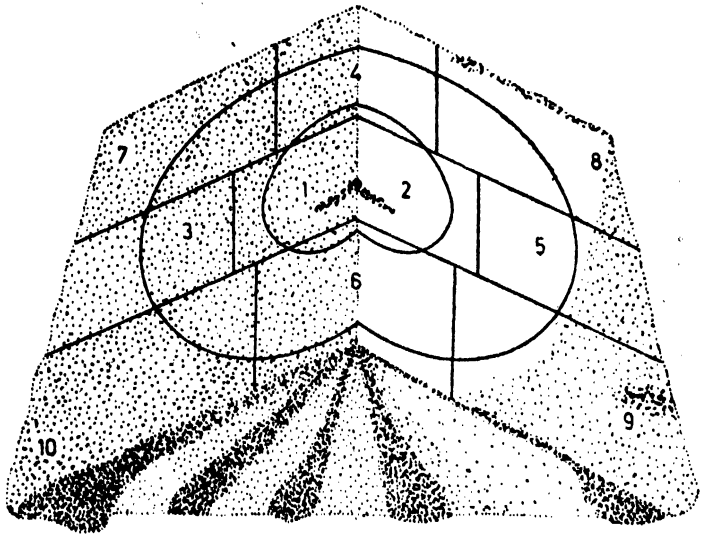


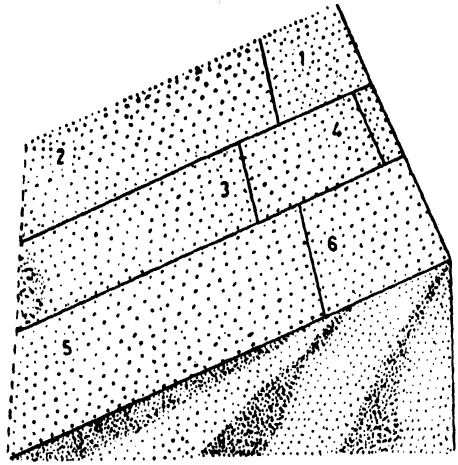
FIG. 5 SAMPLING PATTERN FOR UNDIVIDED WAGONS OR TRUCKS



Withdraw One Vertical Core of Material from Each Numbered Location.

6A Sample Points for Coned or Ridged Pile

FIG. 6 SAMPLING PATTERN FOR BINS AND PILES — *Contd*



Withdraw One Vertical Core from Locations 1 and 6 and Two Vertical Cores Each from Locations 2, 3, 4 and 5.

6B Sample Points for Half or One-Sided Pile

FIG. 6 SAMPLING PATTERN FOR BINS AND PILES

3.4.1.2 For sampling material with uniform stream flow of three minutes or more, such as a transfer or shipment from a bin or large holding hopper, or for stream sampling from a continuous production unit, a minimum of 10 equally timed and spaced stream cuts should be taken during the transfer operation. Avoid sampling of trickle, fines or dust.

3.4.1.3 For sampling material from a blender or other batch unit which has only short periods of material flow, take a minimum of 10 stream cuts from a spout or at the end of the transfer belt. Take one or more stream cuts for each batch, but vary the moment of the stream cut on consecutive batches. For example, the first batch might be sampled early in the batch discharge, the second near the middle of discharge, the third batch towards the last third of the discharge. Then repeat the collection intervals, or alter to provide more intervals, or more frequent sampling.

4. COMPOSITING AND SAMPLE REDUCTION

4.1 Sample increments obtained from bags as described under **3.1** or from wagons as described under **3.2**, or from piles as described under **3.3** or from belt under **3.4** shall be mixed together thoroughly to form a composite gross sample. This is reduced to about 0·5 kg using a riffle as described under **4.2** or by coning and quartering as described under **4.4**, if a riffle is not available.

4.2 Riffle with Four Pans

4.2.1 The riffle (*see* Fig. 7) shall be of corrosion resistant material and so designed that uniform feeding of sample material, at the top of the rectangular openings, divide the sample into representative halves. The size of the riffle shall be appropriate to the quantity of sample being reduced. Pans must fit top of riffle from end to end of partitioned section.

4.2.2 For most unground fertilizer samples, the chute opening shall be 1·0 to 1·5 cm.

4.3 Reduction of Sample

4.3.1 The following basic steps shall be observed while using riffle for reduction of sample.

4.3.1.1 Clean the equipment thoroughly for any adhering particles.

4.3.1.2 Set riffle level, not tilted in any direction.

4.3.1.3 Place two empty pans in position beneath the riffle.

4.3.1.4 Transfer the collected sample to one or two of the remaining pans, as required. Each pan shall be not more than two-thirds full. Level the surface of the sample in the pan before continuing.

4.3.1.5 Using both hands, position the pan containing the sample length-wise over the riffle as near the centre as possible, at right angles to the partitions.

4.3.1.6 Tilt the pan to let the material flow evenly into the riffle in a continuous stream, but not fast enough to flood the riffle. Collect entire sample in the pans beneath riffle.

4.3.1.7 Place two empty pans beneath the riffle and repeat the procedure as given in **4.3.1.4**, **4.3.1.5** and **4.3.1.6** at least twice to mix the sample thoroughly.

4.3.1.8 If required, repeat **4.3.1.4** through **4.3.1.6** with the material in one of the two pans below the riffle, until sample is reduced to approximately 0·5 kg. (If desired, retain the second half as reserve, until the preparation is completed on the portion to be analyzed.)

4.3.1.9 Transfer final sample to moisture proof container, and mark for identification.

NOTE — The container must be large enough to hold the entire final sample. None should be discarded. If the only available container is too small, sample may be reduced by riffling twice and saving one-half at each step, resulting in three-fourths of the original sample.

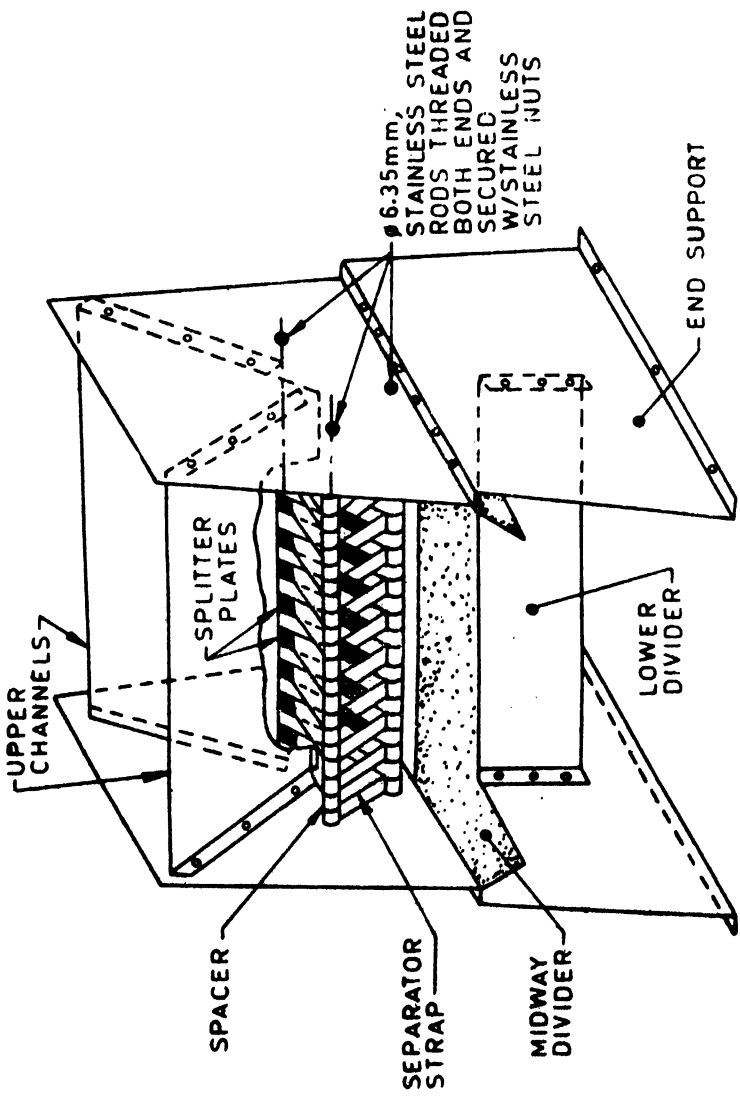


FIG. 7 STRUCTURE OF SAMPLE RIFFLE

4.3.1.10 If an unground portion of sample is to be supplied to another laboratory for check analysis, the unground portion sent shall be 0·25 kg or more.

4.3.1.11 Clean equipment thoroughly before storing or reuse.

4.3.2 *Coning and Quartering*

4.3.2.1 If coning and quartering are adopted, the following procedure shall be followed.

4.3.2.2 Place the gross sample on a clean polythene or other flexible sheet. Roll the sample slowly into a pile in the centre of the sheet and then spread it gently into a flat circle with a smooth flat surface.

4.3.2.3 Using a thin flat edge, separate the circle into two halves along any diameter. Using the edge, increase the diameter line by about 6 mm by carefully pushing the material in each half away from the centre.

4.3.2.4 Mark another diameter at right angles to the first and repeat the procedure as given in **4.3.2.3**.

4.3.2.5 With a scoop remove two opposite quarters from the sheet. The remaining two quarters on the sheet are mixed well. Repeat the procedure till the sample is suitably reduced but not less than 0·5 kg. Any further reduction should be carried out only after grinding the sample.

5. PREPARATION OF SAMPLE

5.1 Grind the entire sample in a glazed porcelain mortar pestle or mechanical grinder after reduction under **4.3**.

5.2 Grind fertilizer mixtures to pass through 355 microns. For fertilizer materials and moist fertilizer mixtures which form a paste on putting pressure, grind in porcelain pestle and mortar (*see* Note) to pass through 1 mm IS Sieve. For dry mixtures which tend to aggregate, grind in a porcelain pestle and mortar (*see* Note) to pass through 355 microns IS Sieve.

NOTE — For hygroscopic fertilizers, it is preferable to use electric mixer. In case of fertilizers like urea, ammonium sulphate and muriate of potash, grinding is not necessary.

5.3 For moisture determination in granular material the samples shall be suitably ground.

5.4 Grind as rapidly as possible to avoid loss or gain of moisture during the grind operation, but avoid over-heating the sample.

5.5 Place entire sample on flexible rolling sheet of glazed paper or smooth oil cloth for mixing.

5.6 Roll sample slowly from four directions until sample has been thoroughly mixed (10 times is usually considered adequate). This ensures complete mixing. Trying to roll too rapidly will cause a sliding.

5.7 Spread the sample flat and transfer about 200 g of sample to a 250 ml wide mouthed air tight container.

5.8 Label the container properly.

6. SAMPLING FOR ANHYDROUS AMMONIA

6.0 Outline of the Method — The sampling method of anhydrous ammonia is based on the assumption that the material to be sampled contains only a small amount of impurity primarily water (about 0·2 percent). Duplicate samples shall be taken from each tank or vessel sampled.

6.1 Apparatus

6.1.1 *Weathering Flask* — 2 liter capacity graduated from 0 to 10 ml and made of heat resistance glass (*see* Fig. 8).

6.1.2 *Stopper* — Rubber with bend tube bent as shown in Fig. 9.

6.1.3 *Sampling Line and Connection Assembly* — (*see* Fig. 10) in which flexible steel sampling hose 1·2 m is shown with 6 mm NPT coupling at one end and about 3 mm insulated steel tubing delivery tip at the other end.

6.2 Reagents

6.2.1 *Charcoal* — Analytical reagent grade and passing through 14 to 20 mesh.

NOTE — If the sample is expected to contain excessive amount of water (one percent or more), one piece of the charcoal may be added to each tube before introduction of the sample.

6.3 Procedure

6.3.1 Take two dry, clean weathering flasks. Connect the sampling line connection assembly to the unloading valve of the tank, vessel, or line to be sampled. Open the valves slowly and purge the sampling line connection assembly thoroughly by venting 3 to 4 litres of ammonia. Close the sample line glove valve. Remove the vented stoppers from the flask and insert the adapter end of the sampling line connection assembly. Open the sample line valve and slowly fill the weathering flask to the 1 800 ml mark, close the sample line valve. Remove the sampling line adapter and insert the vented stopper in the weathering flask. Repeat as above and fill the second flask. Close tank discharge valve and

remove the sampling line connection assembly. Note the pressure in the vessel, container, or in the line of material sampled. Tag the collected samples for identification and submit to the laboratory for processing.

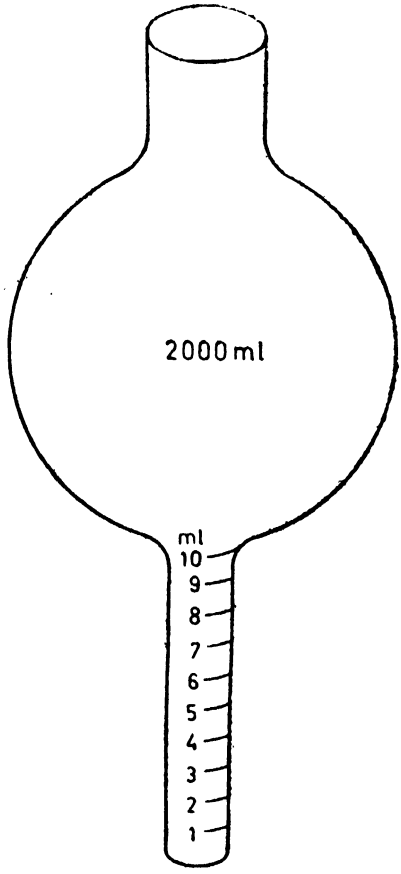


FIG. 8 WEATHERING FLASK

6.3.2 The gauge pressure of the container shall be read at the time of sampling to determine the evaporation factor. The evaporation factor for different pressure in vessel is given in Table 3.

6.4 Precautions

6.4.1 Ammonia is extremely caustic inflammable gas. It reacts violently with strong oxidising agents, halogens and strong acids and

forms explosive compounds with many heavy metals such as silver, lead, zinc, mercury and their salts, especially with halides.

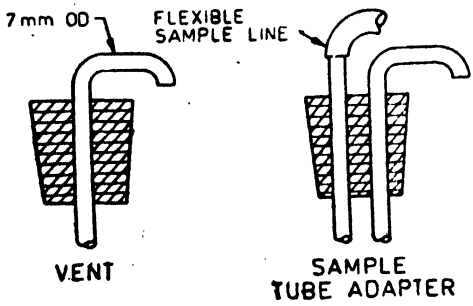
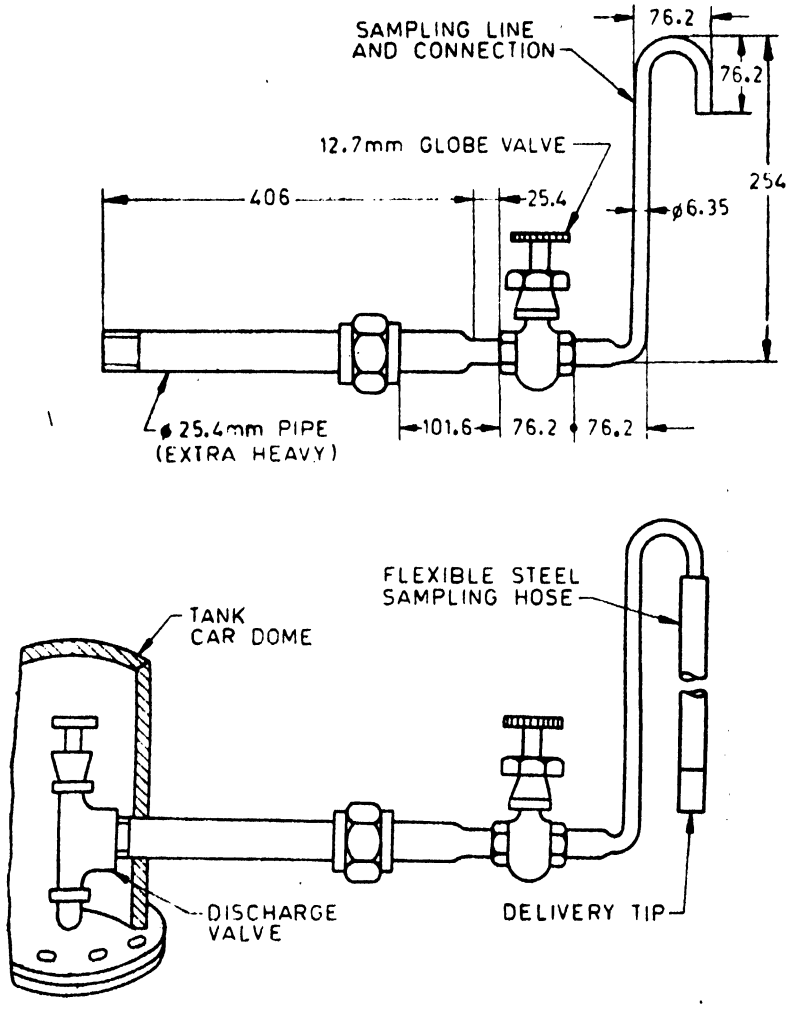


FIG. 9 WEATHERING FLASK ADAPTER

TABLE 3 EVAPORATION FACTOR FOR DIFFERENT PRESSURE IN VESSEL

(*Clause* 6.3.2)

PRESSURE IN VESSEL IN /kg cm² GAUGE	EVAPORATION FACTOR (*F*)
(1)	(2)
0·0	1·000
0·703 0	0·963
1·406 1	0·940
2·103 2	0·920
2·812 2	0·900
3·515 3	0·885
4·218 2	0·870
4·921 4	0·860
5·824 6	0·850
6·327 6	0·840
7·030 7	0·830
7·733 8	0·821
8·436 8	0·813
9·139 9	0·805
9·913 3	0·797
10·546 0	0·789
11·249 1	0·782
11·952 2	0·776
12·635 3	0·770
13·358 3	0·764
14·061 4	0·758



All dimensions in millimetres.

FIG. 10 SAMPLING LINE AND CONNECTION ASSEMBLY

6.4.2 Liquid anhydrous ammonia causes severe burns on contact. Ammonia gas in concentrations of 6 000 to 10 000 ppm (by volume) is lethal within few minutes. Irritation of the eyes, respiratory tract and

throat result from concentrations as low as 500 to 1 000 ppm, a concentration of 2 000 ppm produces conclusive coughing and may prove fatal after a short exposure which may be less than half an hour. The maximum concentration tolerated by the skin for more than a few seconds is 2 percent that is when suitable respiratory protection is worn. The maximum allowable concentration for 8 hours working exposure is 50 ppm.

6.4.3 Use skin, eye and respiratory protection when handling anhydrous ammonia. Rubber or other non-porous gloves, offering complete protection to the hands and lower arms must be worn while sampling anhydrous ammonia. Full coverage goggles must also be worn to protect the eyes unless an approved gas mask is used. The gas mask need only be used if sampling cannot be done without possible inhalation of vapours.

6.4.4 Avoid contacting liquid ammonia. In case of contact, immediately flush the affected part with plenty of water for at least 15 minutes. Get medical attention at once in case of burns especially to the eyes, nose and throat or if, the victim losses consciousness.

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